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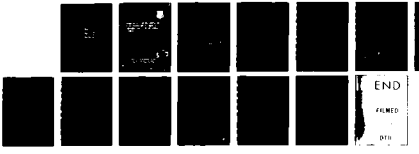

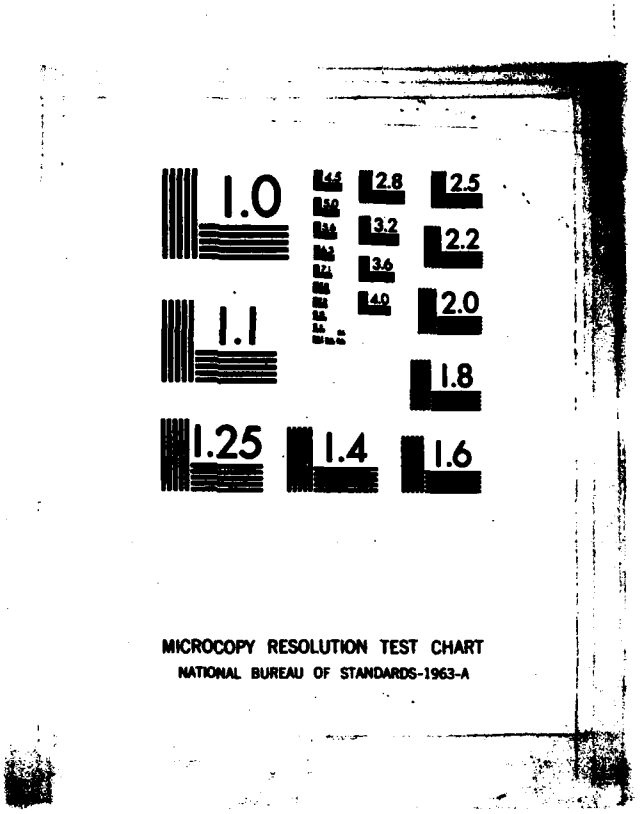

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RADC-TR-84-144
In-House Report
June 1984

FORMATION OF A LIMITING COMPOSITION IN THE PREPARATION OF TERNARY III-V SEMICONDUCTORS BY THE VPE-HYDRIDE TECHNIQUE

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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

A study was undertaken to improve the quality of epitaxial layers of $Ga_xIn_{1-x}As$ semiconductors by adding a continuous hydrogen chloride (HCl) etch to the mixing zone of the reactor in the vapor phase epitaxy (VPE)-hydride technique. An unusual observation was noted when the partial pressure of HCl in the mixing zone was equal to or greater than ~~7×10^{-4}~~ atm in the present study. At these partial pressures of HCl, only one ternary is formed and has a composition corresponding to $Ga_{0.47}In_{0.53}As$. This phenomenon is explained with a mechanism based on the Langmuir's adsorption isotherm, where saturated HCl absorption occurs. The study shows that the net growth rates of layer formation changes from positive to negative values with increasing pressures of HCl in the mixing zone.

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Preface

During the course of this study, the authors have received assistance from many others. They wish to acknowledge the help provided by Jane A. Horrigan in performing the x-ray analyses of the samples and to Joseph J. Comer for the determination of the thicknesses. The authors gratefully acknowledge the help and advice from John K. Kennedy. Single crystal InP substrates were grown and polished by Dr. Brian S. Ahern.

Contents

Illustrations

Formation of a Limiting Composition in the Preparation of Ternary III-V Semiconductors by the VPE-Hydride Technique

1. INTRODUCTION

The ternary, $Ga_{0.47}In_{0.53}As$, is an important material for applications in the microwave and optoelectronic devices fields. This ternary can be lattice-matched to semi-insulating InP substrates. $Ga_xIn_{1-x}As$ offers several advantages for their use in high microwave frequency FET's, for example, high low-field electron mobility and high peak electron drift velocity.[1] In addition, extensive research[2,3] is being conducted for the development of high quality $Ga_xIn_{1-x}As$ photodetectors for use in optical communications. These detectors fabricated with epitaxial layers of $Ga_xIn_{1-x}As$ are sensitive to the long wavelength region (1.0 to 1.7 μm), where silica fibers exhibit their lowest loss.

(Received for publication 12 July 1984)

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Work has been performed at this laboratory[4] and elsewhere[5,6] where epitaxial layers of $Ga_{0.47}In_{0.53}As$ have been deposited onto InP substrates by the vapor phase epitaxy (VPE) technique using a Ga-In alloy as the source of the group III elements. This research results in the growth of epitaxial layers of $Ga_xIn_{1-x}As$ exhibiting good morphology and having electrical properties that compare favorably to those grown by the other methods, for example, LPE, MBE, and MOCVD. However, microscopic examination of the edge of several beveled samples revealed that defects were formed in the three-μm region of the epilayer nearest the interface with the substrate. This lowers device performance and is unacceptable for device manufacturing. These defects in the epilayer probably result from imperfections at the surface of the substrate caused by incongruent decomposition of InP. This decomposition of the InP substrate probably occurs when the substrate is heated in the deposition zone before the epitaxial layer is deposited.

Recent research[7] at this laboratory has shown that epitaxial layers of InP with good morphology and electrical characteristics could be grown on semi-insulating InP substrates by using a continuous in-situ HCl etch. The method used for these preparations was the VPE-hydride technique using a three temperature zone quartz reactor. The continuous in-situ HCl etch probably removes any impurities and defects that are present on the substrate surface. These impurities and defects hinder good crystal growth of the epitaxial layer. This same approach was extended to the present study where the effect of a continuous in-situ HCl etch on the growth of $Ga_xIn_{1-x}As$ on semi-insulating InP substrates was investigated.

The present paper reports an unusual observation made during this study: the ternary, $Ga_{0.87}In_{0.13}As$, is the only product formed when the partial pressure of the etch HCl is equal to or greater than 7×10^{-4} atm. This phenomenon is explained by a mechanism similar to the one describing surface-catalyzed reactions, which is based on the Langmuir's adsorption isotherm.

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2. EXPERIMENTAL

The preparation of the epitaxial layers of $Ga_xIn_{1-x}As$ was performed in the quartz reactor shown in Figure 1. The quartz reactor has three temperature zones: source, mixing, and deposition. These zones are heated by "clamshell" resistance heaters. The temperatures of the source, mixing, and deposition zones were maintained at 800, 850, and 675°C, respectively, throughout the study. The reactor has three gas inlet flow systems: $AsH_3/HCl/H_2$ (mixing zone entry), HCl/H_2 (source zone entry), and H_2 (forechamber). The flow rates are regulated by Tylan mass flow controllers. The reactant gases are the highest purity products available. Arsine (99.998 percent) was supplied as a 10 percent mixture in hydrogen (H_2) (99.999 percent) from Ideal Gas Products, Edison, N.J. The H_2 carrier gas, supplied by American Industrial and Medical Products, Auburn, Mass., was 99.999 percent pure. The H_2 was further purified by a Hydrogen purifier (Palladium Diffusion Process-Engelhard). Hydrogen chloride was 99.995 percent and supplied by Precision Gas Products, Inc., Rahway, N.J. Indium (99.9999 percent) was obtained from Metal Specialties, Inc., Fairfield, Conn.. Gallium was 99.9999 percent and a product of Alusuisse Metals, Inc., Fairlawn, N.J.. The alloys were prepared in quartz boats and weighed between 99 and 107g. The surface areas of the alloys in the quartz boats were approximately 26 cm^2. The alloys were prebaked for 50 h in an H_2 atmosphere in order to lower the carrier concentrations in the resulting epitaxial layer.[6]

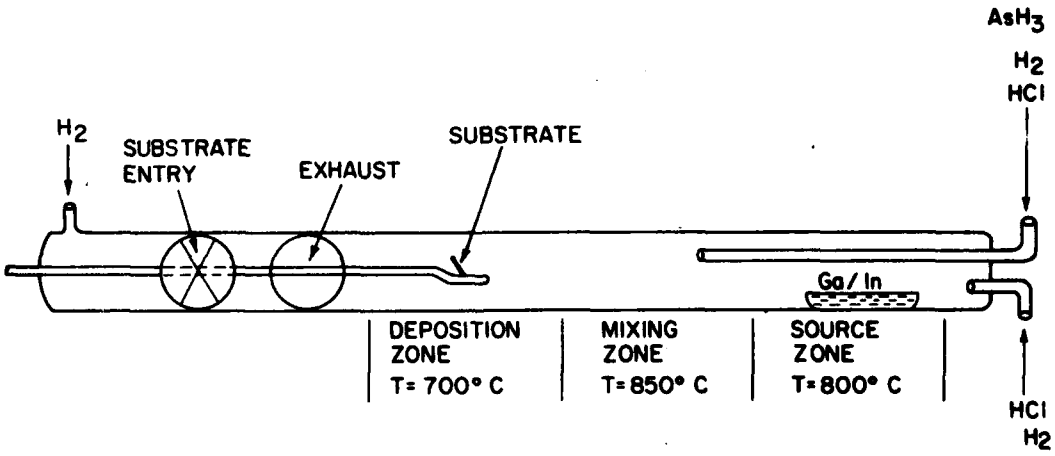

Figure 1. Vapor Phase Epitaxy (VPE)-Hydride Quartz Reactor for the Preparation of Epitaxial Layers of $Ga_xIn_{1-x}As$

The substrates were prepared from a liquid-encapsulated Czochralski (LEC)-grown InP(Fe-doped) boule. Slices of the boule were cut 3^{o} off the (100) plane towards the (111) plane. Slices were polished on an Electropolisher (Sylvania Co.) with Pellon Pan W pads (J.I. Morris Co., Southbridge, Mass.) using a 0.5 to 1.0 percent bromine-isopropyl alcohol solution. The substrates were degreased by treatment with toluene, 1,1,1,-trichloroethane, and acetone. The substrates were etched for 5 min in Caro's acid (1 water + 1 H_2O_2 + 5 H_2SO_4). After washing with water, the substrates were further etched for 2 min in a 0.3 percent Br_2-methanol solution and then rinsed with methanol. This was followed by another 5-min Caro's acid etch. The substrates were blowndried with nitrogen (N_2).

The procedure used to grow the ternaries on InP (Fe-doped) substrates in the present study was the following: the holder with the substrate was placed in the forechamber (H_2 ambient) with the substrate entry stop-cock closed. The H_2 flow rates to the source and mixing zone inlets were 340 cc/min for a total of 680 cc/min. After 15 min, the substrate entry-cock was opened and the two H_2 flow rates were adjusted to 445 cc/min, the 10 percent arsine flow rate was set at 100 cc/min (arsine = 10 cc/min), and the etch HCl flow rate to the mixing zone was adjusted to the designated value of the experiment. The flow rate values for the etch HCl ranged from zero to 5 cc/min. After 5 min, the substrate was positioned in the deposition zone and the source HCl flow rate fixed at 2.3 cc/min. This was time zero for the reaction time. The reaction time was usually 60 min. The partial pressures of the arsine, H_2, and source HCl were maintained at these values throughout the study.

The compositions of the grown ternaries of $Ga_xIn_{1-x}As$ were determined from Vegard's law, where lattice constants are plotted as a function of composition. The lattice constants were determined by x-ray diffractometry with Cu$K\alpha$ irradiation using InP as an internal standard. Microprobe analyses (SEM/EDAX) verified this method of determining the compositions of the epitaxial layers.

Net growth rates were determined from the surface area of the substrate, mass of deposit, density of the ternary, and time duration of growth. The values obtained were halved since essentially equal amounts of the ternaries grew on both sides of the substrate. The thicknesses of the ternary layers were measured from scanning electron micrographs of the cross-sections of epilayers on the substrates.

3. RESULTS AND DISCUSSION

This study was initiated to observe whether high-quality epitaxial layers of $Ga_{0.47}In_{0.53}As$ could be achieved when a continuous HCl etch was added to the mixing zone of the quartz reactor shown in Figure 1. A series of experiments were performed by adding increasing amounts of HCl to the mixing zone of the VPE-Hydride reactor. The compositions of the ternaries obtained in these experiments are shown graphically in Figure 2. The Ga concentration in the epilayers increases linearly when the partial pressures of HCl in the mixing zone is increased. An unusual phenomenon is noted when the partial pressure of the HCl in the mixing zone is equal to or greater than 7×10^{-4} atm. Over this range of HCl-etch partial pressures, only one composition of $Ga_xIn_{1-x}As$ is formed and corresponds to $Ga_{0.87}In_{0.13}As$. Even excessive amounts of HCl etch, for example, 5×10^{-3} atm, does not change the composition of the ternary. A mechanism for this unique and remarkable observation is given below. When no HCl is added to the mixing zone at these parameters, the $Ga_{0.47}In_{0.53}As$ is

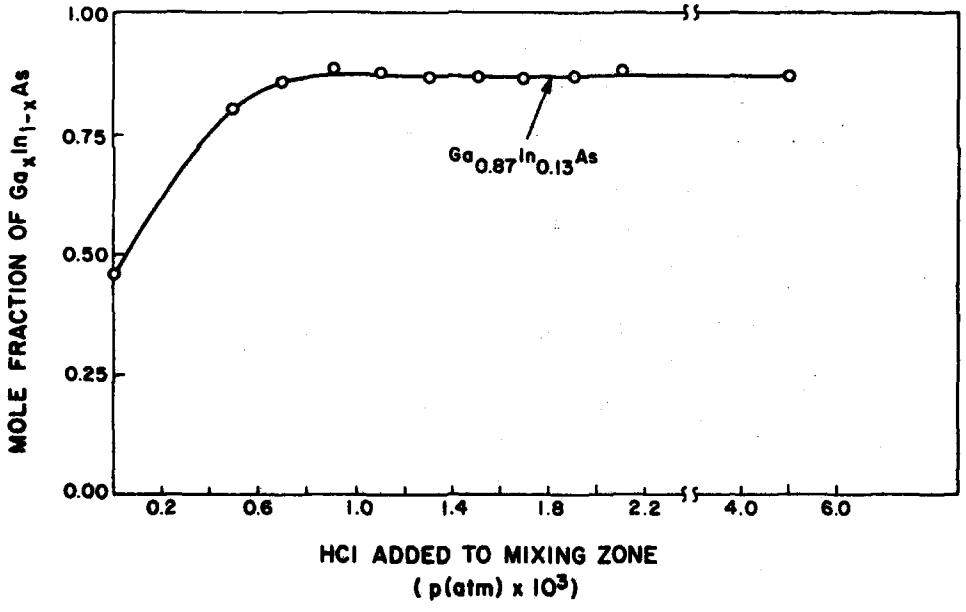

Figure 2. Compositions of Ternaries, $Ga_xIn_{1-x}As$, Prepared with Various HCl Partial Pressures in the Mixing Zone. Hydrogen Chloride (source) partial pressure, 2.3×10^{-3} atm; AsH_3 partial pressure, 1.0×10^{-2} atm; total H_2 flow rate, 980 cc/min. Source alloy composition, 88.2 mole percent In and 11.8 mole percent Ga. Substrate temp = 675°C; mixing temp = 850°C; and source temp = 800°C. Reaction time = 60 min

formed. The surface of this epitaxial layer had many minor hillocks and etch pits with some highly concentrated defects areas. The quality of the surfaces of the epitaxial layers grown with a HCl etch were improved and exhibited fewer defects. A very smooth surface with few hillocks and etch pits was obtained when the partial pressure of the HCl was 7×10^{-4} atm.

A study of the net growth rate of $Ga_xIn_{1-x}As$ as a function of the partial pressure of mixing zone HCl was determined and the results are shown in Figure 3. The growth rates increase to a maximum at 9.0×10^{-4} atm and then decrease to negative values with increasing etch HCl. The observed maximum has been previously discussed in the publications of Erstfeld et al,[7] and Mizutani

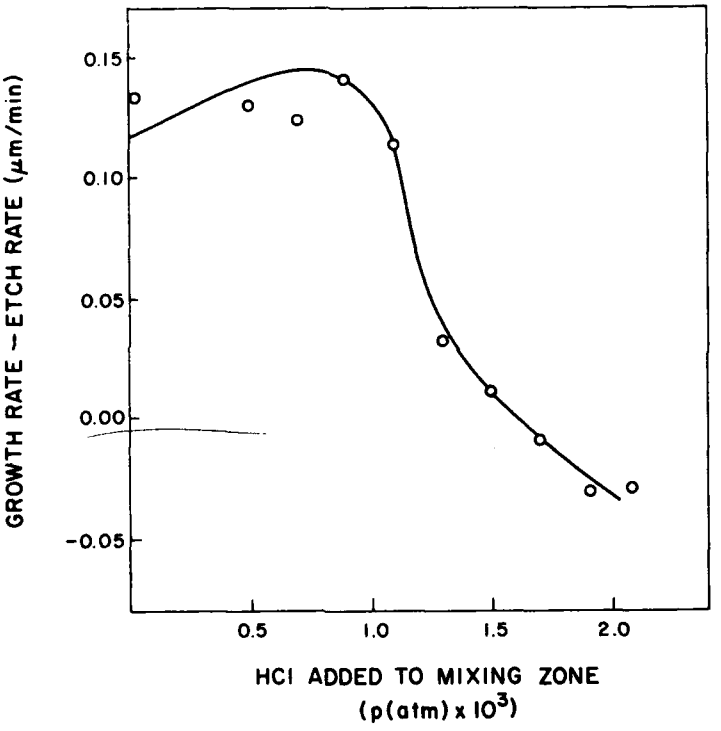

Figure 3. Net Growth Rates of Ternaries as a Function of HCl Partial Pressures in the Mixing Zone. Hydrogen Chloride (source) partial pressure, 2.3×10^{-3} atm; AsH_3 partial pressure, 1.0×10^{-2} atm; total H_2 flow rate, 980 cc/min. Source alloy composition, 88.2 mole percent In and 11.8 mole percent Ga. Substrate temp = 675°C; mixing temp = 850°C; and source temp = 800°C. Reaction time = 60 min

and Watanabe.[8] Figure 3 shows that even though $Ga_{0.87}In_{0.13}As$ was being deposited, the net growth rates were negative for partial pressures of 1.6×10^{-3} atm or higher for the etch HCl. The negative growth rate is the result of greater amounts of the substrate, InP, being etched than the amount of $Ga_{0.87}In_{0.13}As$ being deposited. Epitaxial layers of $Ga_{0.87}In_{0.13}As$ with thicknesses of 2.0 and 1.9 μm were obtained when the etch HCl partial pressures were 1.5×10^{-3} atm and 1.9×10^{-3} atm (negative growth rate region), respectively.

The formation of $Ga_{0.87}In_{0.13}As$ in the above study can be explained by the following mechanism: the InP substrate is etched by the HCl in the mixing zone before the mixture of As_2, As_4, InCl, GaCl, and H_2 and the substrate reaches equilibrium, where deposition occurs. The kinetics of these reactions have been shown to be relatively slow.[9,10] After equilibrium saturation, deposition of the ternary takes place. This segment of the mechanism was verified by reducing the reaction time from 60 min to 10 min. The results are shown in Figure 4 and depict a more negative net growth rate (-1.03 μm/min) at 10 min than that obtained at 60 min (-0.03 μm/min). Hydrogen chloride molecules are then adsorbed onto the forming epitaxial layer and react preferentially with InAs. Thermodynamic calculations show that the etching of InAs is more favorable than GaAs.[11,12] These reactions result in a ternary layer with less In than that obtained when no etch HCl is present in the mixing zone. The amount of In removed increases with increasing partial pressures of etch HCl. Above 7×10^{-4} atm, the amount of InAs removed is constant and results in the formation of $Ga_{0.87}In_{0.13}As$ over a wide range of HCl partial pressures. At these higher HCl pressures, the surface of the $Ga_xIn_{1-x}As$ is saturated with reactant molecules (HCl) and increasing the pressure does not increase the number of reactant molecules adsorbed. Consequently, the amount of InAs removed remains constant. This mechanism is similar to the one describing surface-catalyzed reactions based on the Langmuir's adsorption isotherm.[13]

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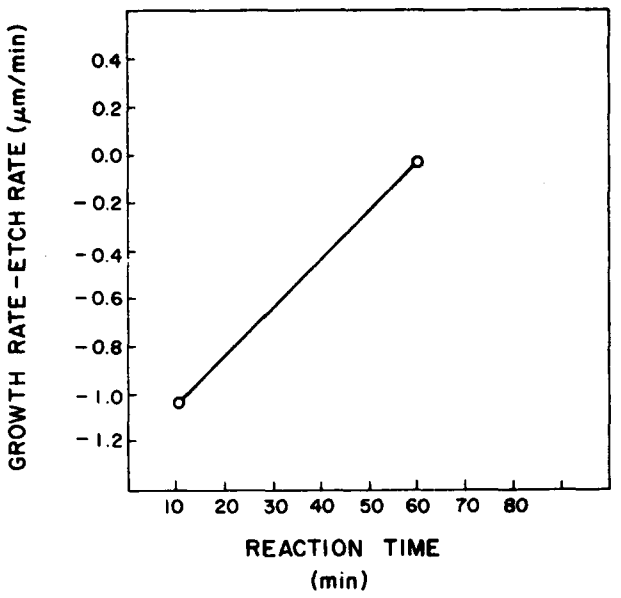

Figure 4. Net Growth Rates as a Function of Reaction Time. Hydrogen chloride partial pressure in the mixing zone, 1.9×10^{-3} atm; HCl (source) partial pressure, 2.3×10^{-3} atm; AsH_3 partial pressure, 1.0×10^{-2} atm; total H_2 flow rate, 980 cc/min. Source alloy composition, 88.2 mole percent In and 11.8 mole percent Ga. Substrate temp = 675°C; mixing temp = 850°C; and source temp = 800°C

The electronic properties of these layers have not been investigated, but this method of preparation offers the possibility of reducing the effects of lattice mismatch between a binary and ternary epitaxial layer. Further studies will also be undertaken to determine whether a constant deposition of $Ga_{0.47}In_{0.53}As$ can be achieved when the gas mixture in the mixing and deposition zones contain less Ga.

4. CONCLUSION

The addition of HCl to the mixing zone of a VPE-Hydride reactor has a mark effect on the composition of the ternary, $Ga_xIn_{1-x}As$, produced. When the partial pressure of the HCl etch is equal to or greater than 7×10^{-4} atm, only one composition of the ternary is produced and corresponds to $Ga_{0.87}In_{0.13}As$ at the parameters of the present study. These results show that this ternary can easily be prepared by adding HCl to the mixing zone of a VPE-Hydride reactor. The net growth rate of the formation of $Ga_{0.87}In_{0.13}As$ changes from positive to negative values with increasing partial pressures of the HCl etch. A mechanism based on the Langmuir's adsorption isotherm is suggested to explain this new phenomenon.

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